MML TR-93-17

Composite Panel Failure Analysis
and Improvement Plan

Prepared by:

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Prepared for:
U.S. Army Belvoir Research, Development
and Engineering Center
Fort Belvoir, VA 22060-5606

Under Contract No. DAAK70-90-C-0019

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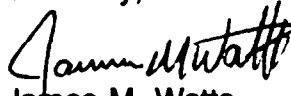FAX: (410) 204-2100

January 5, 1994

US Army Research Development & Engineering Center
Attn: Mr. Brian Hornbeck STRBE-JBC
Ft. Belvoir, VA 22060-5606

Subject: Contract DAAK-70-90-0019, Final Report

Dear Mr. Hornbeck: ·

In accordance with the requirements of the above subject contract, enclosed please
find two (2) copies of our Final Report "Composite Panel Failure Analysis and
Improvement Plan" and two (2) copies of the Monthly Cost Reports for the period
ending November 28, 1993. We are also enclosing a completed DD250 Thank you.

Sincerely,

James M. Watts
Contracts

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Engineering test specimens and prototype XM19 Landing Mat sections were examined to determine the reasons for below-standard performance. Electron microscopy, x-ray photoelectron spectroscopy and thermal analyses were used in addition to visual microscopy, to determine the locus of failure, residual adhesive cure, and identify contributing causes of the failures. A number of poor production practices were identified, and a prototype porcess improvement plan proposed.

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1.0 Introduction

Composite panel medium duty aluminum landing mats developed by the Bridge Division of the Belvoir Research, Development and Engineering Center have exhibited failures in proof and quality conformance testing. Failure analysis of test specimens and a type I panel after rollover testing was performed in order to determine the causes and locations of the failures. Visual inspection, scanning electron microscopy (SEM), and x-ray photoelectron spectroscopy (XPS) were employed to determine the precise locus of failure in each case, and to examine the potential causes. Differential scanning calorimetry (DSC) was used to study the cure state of adhesive taken from various regions of the test specimens, and was compared with similar adhesive run through representative cure schedules. The results of these analyses clearly identify a number of construction problems. After review of the fabrication process specifications, a number of recommended amendments are discussed.

2.0 Visual Inspection

Two types of test specimen were provided by the Bridge Division, short beam shear and static edge member. Short beam shear specimens consist solely of upper and lower face sheets and honeycomb separator. Static edge member panels also feature a portion of the edge extrusion along one side of the specimen. Table I lists the short beam shear specimens that were examined, and Table II lists the static edge member specimens .

Table I. Short beam shear test specimens

#	Sample ID	Failure type	Apparent cause
1	1 - 1240	extensive face sheet delamination	poor filleting
2	Ext 1 - 5440	cell collapse, front sheet delamination	adhesive slick-off
3	2A	v-notch	
4	2A 6620	v-notch	
5	2B - 6580	v- notch	
6	2C - 5520	cell collapse, back panel delamination	
7	3	extensive face sheet delamination	severe bridging
8	6 - 5260	cell collapse, back panel delamination	

Table II. Static edge member shear test specimens

#	Sample ID	Failure appearance
1	1A	no edge adhesive
2	1B - 6520	severe bridging at edge, extrusion cocked
3	1C - 4480	no edge adhesive, some bridging
4	1D - 9700	no edge adhesive
5	1E - 3240	bridging and poor filleting of honeycomb
6	1F - 4400	insufficient adhesive and bridging on honeycomb near edge

The first step in failure analysis was visual inspection of each test specimen to identify possible causes of failure and focus on regions of the specimens for more detailed analysis. The skin on the side of the specimen that appeared to have failed adhesively was peeled away in order to allow visual inspection of the entire bonded region. Figures 1-6 are macrographs of the short beam specimens after skin removal. Some problems are immediately apparent. Sample #7 (Figure 1), for example, shows no evidence that the core ever contacted the face sheet during bonding. This situation is caused by lack of pressure over the region of the bondment in question, and is frequently seen in large bondments cured in platen presses. If the platens are not perfectly flat, or if the bondment itself is not perfectly uniform, high points exert significant pressures but low points are "bridged", and do not maintain contact. Sample #1 (Figure 2) shows poor filleting of the adhesive on the edges of the honeycomb core, which is probably a less severe case of bridging. When the core is not firmly pressed into the adhesive, flow along the side of the core is minimized, and a poor bond results. In both cases, the lack of bonding led to very low strength failures in testing, as indicated by the lack of deformation of the core, and the small deformation of the face sheets. Higher strength bondments suffer more extensive deformation before stressing the bond sufficiently to cause failure.

Sample #2 (Figure 3) shows a failure between the adhesive and the face skin in the test region. This failure mode appears infrequently in the test specimens studied. Such failures are referred to as "slick-offs", and are generally caused by poor surface preparation or by surface contamination prior to bonding.

The other short beam samples have less obvious failures. They are characterized by deformation of the honeycomb core, sometimes confined to the top surface directly under the bend test pressure point ("v-notch"), and sometimes at a 45° angle through the core at varying distances from the center of the specimen. Figures 4 and 5 show v-notch short beam specimens, and Figure 6 shows a 45° crushed core specimen. What these specimens seem to have in common is a region where the adhesive has been pulled off the core walls, leaving adhesive "stalactites" on the face sheet. Note that the normal overstress failure mode leaves the adhesive fillet attached to the core, and a fractured adhesive on the face sheet. The location of the debond region governs the type of failure

seen in the test specimen. When this disbond occurs at the top sheet the result frequently is the v-notch behavior. When the bottom sheet pulls loose, the 45° crush pattern results.

Core disbonds can be caused by contamination on the cell walls. However, most of the debonds seen on these specimens have an unusual feature in common. Figure 7 shows a side view macrograph of a cell wall taken from short beam specimen #4. Note that the adhesive fillet is not uniform in thickness across the cell. This pattern seems to persist throughout the region of core pull-out. Where the fillet is very small the applied stress necessary to initiate a failure is also small. Depending on the stress state of the bond, this can lead to bending of the cells, and a peel stress being applied to the thicker fillet, leading to pull-off. Non-uniform wetting of the core is unusual in my experience. The typical pattern seen in the short beam specimens is of a thin band or arc of asymmetric cells. These region probably represents an area where the core was slightly thicker, and was under higher stresses during cure, causing deformation that led to non-uniform wetting.

Static edge member test specimens show two primary failure modes. In all cases there is insufficient skin adhesive over the rails at the edge of the specimen. Thus the extrusion pulls out under stress. This is evident in photographs taken of the specimens shown in Figures. 8-13. In many cases a residue of core to rail adhesive is seen on the skin to rail bonding surface, since no skin adhesive was present to seal off this area from the expanding core to rail adhesive. Note that core splice adhesive is not suitable for structural bonding between aluminum sheets, and in no way can substitute for skin to rail adhesive. A particularly blatant example in shown in Figure 10, where the skin adhesive stops at least 1 in away from the edge of the core.

The other failure mode seen in these specimens was obvious bridging, leading to poorly bonded core. Figure 9 shows static edge specimen #2, where no core bond existed about 3 in. away from the rail. In this case the rail extrusion was cocked, and the tilted rail kept the face sheet from contacting the honeycomb. Evidence of bridging, i.e., lack of core imprinting on the face sheet adhesive, can be seen in all cases except static edge sample #4.

The type I rollover failure panel shows all the problems seen on the test specimens, and more. Figures 14 and 15 show the bottom skin and opposite core and rail frame respectively after peeling the skin off the panel. Note the large region in the center of the panel where the (grey) adhesive has been pulled off the (shiny and therefore white) skin. The generally bare regions around the edges of the skin indicates the lack of adhesive over the rails. The core side shows extensive core delamination at one edge of the panel. A closer view of this region (Figure 16) shows an unbonded rail, and an unusual pattern on the honeycomb adjacent to the split honeycomb core region. The skin side view (Figure 17) shows a large gap in adhesive at the rail edge of this region (arrow), and a very poor cell imprint in the adhesive (bridging) in this spot. Finally there is a region (shown in Figure 18) below the bare spot in the center of the skin where the adhesive is dark brown, and the support material seems to have vanished (melted?) that suggests a severe hot spot in the platen.

The large debond region in the center of the panel was the main cause of the large scale buckling it suffered. But the lack of edge support, and poor core bonding at the edge where rollover started was the cause of core splitting. Finally the burn mark in the skin adhesive shows a significant problem that also could cause inadequate core to skin adhesion and panel failure.

3.0 Surface Analysis

3.1 SEM

SEM examination of selected bonding surfaces was undertaken to see if a precise locus of failure could be determined, and to investigate the nature of the surfaces of the face sheets and honeycomb in regions that exhibited poor bonding. Table III describes the specimens that were examined by SEM, and XPS. Identical samples were taken side-by-side for the two surface analysis techniques so that the chemical and morphological results could be directly correlated. In all cases each specimen had two parts; one side was apparently "bare" metal and the other was the adhesive surface that had been in contact with the first. These specimens were picked to represent the regions where potential contamination might be suspected, i.e., where slick-off or pull-off had occurred. One sample, short beam #4, was used as a standard for a nominal bonded surface. A piece of the skin was sharply bent to initiate a adhesive to skin disbond that could be expected to be clean.

Table III. Surface analysis specimens

Sample #	Specimen ID	Description
1	Short Beam #2 (Ext 1 5440)	skin to adhesive slick-off
2	Short Beam #4 (2A)	forced debond between skin and adhesive
3	Short Beam #4 (2A)	core pull-off
4	Static Edge #2 (1B 6520)	top skin to rail slick-off
5	Static Edge #5 (1E 3240)	bottom skin to rail slick-off
6	Type I Panel (58)	skin to adhesive slick-off

The electron micrographs of the surface analysis #1 specimen show a clean, complete break between the metal and adhesive side of the failure, Figures 19 and 20. In this case the metal side of the specimen shows distinct surface features, that are clearly replicated in the adhesive on the opposite side. The surface morphology is typical of an acid etched aluminum, with a pockmarked appearance. This surface is not typical of aerospace bonding practice, for it lacks the porous oxide layer that is receptive to adhesive infiltration and interlocking. This surface treatment should provide somewhat inferior dry bond strengths, but significantly reduced performance under high humidity conditions.

The core disbond (surface analysis #3) shown in Figures 21 and 22 shows typical conversion coating morphology expected on honeycomb aluminum core. The surface is obviously quite smooth, offering little opportunity for the adhesive to form a tight grip. However, this is the same morphology that has proven adequate in hundreds of aerospace bonding applications. XPS results are therefore necessary to indicate whether contamination was involved in the disbond, or whether it was simply a stress overload.

The surface morphologies seen on the other four specimen surfaces are essentially identical, Figures 23-26.. In these cases the same pockmarked metal-side surface is seen, but the features are less distinct. The smoother edges, and slightly bumpy surfaces indicate that an extremely thin layer of adhesive is still clinging to the metal surface, and the failures in theses cases are actually entirely within the adhesive (cohesive), rather than strictly interfacial.

3.2 XPS

X-ray photoelectron spectroscopy is a technique for determining the elemental composition of extremely thin surface layers, nominally just a few atomic layer thick. Thus it is an extremely sensitive way of probing for contamination, and determining the precise locus of failure in bonded specimens.

The results of XPS surveys of the metal sides of adhesive disbonds from the surface analysis specimens are given in Table IV. A clean aluminum surface should only show aluminum and oxygen, but atmospheric carbon contamination is always seen on samples that have been exposed to normal air. In addition, alloying elements in the underlying substrate material also are seen through thin oxide layers. Finally, some trace of materials in etch and rinse baths is frequently detected. Thus a freshly acid etched aluminum surface would have about 20 weight % Al, 45 % O, 30 % C, 3-6 % N, and traces of Cu, Mg, Fe, P (from the etching solution). Sample #2 (a forced skin to adhesive debond) most closely matches this pattern. There are a number of additional trace elements found on this surface that suggest tap water contaminants, including F, Si, Ca, Na, and Cl. Si cou'1 be related to silicates (harmless) or silicone (extremely detrimental). F is a potential problem, since it tends to react with water vapor to produce HF which can attack the surface oxide film and cause bonding problems. Deionized water is recommended for etch solution makeup and rinse bath water for this reason.

The other five specimen surfaces show varying degrees of additional C coverage, presumably due to a retained adhesive film. In particular, #3 (honeycomb edge of short beam sample #4) and #4 (rail to adhesive "slick-off", static edge sample #2) indicate a significant coverage by a C containing film. The other specimens, including #1 ("slick-off" region of v-notch specimen short beam #2), #5 (lower skin to adhesive, static edge #5), and #6 (type I panel skin to adhesive "slick-off"), all show nearly clean aluminum oxide surfaces, indicating interfacial adhesive failure.

Table IV. XPS surface concentration results from metal sides of surface analysis specimens (weight %)

Element	#1	#2	#3	#4	#5	#6
Al	12.9	15.7	0.8	1.7	9.6	12.6
C	40.4	29.4	69.0	66.9	40.2	33.0
O	35.6	39.3	19.2	22.2	34.5	36.8
N	6.3	5.3	7.7	3.9	5.8	9.4
F	1.0	0.9	0.4	0.3	0.9	5.2
Si	1.2	5.0	1.5	3.5	3.3	0.6
Ca	0.9	1.0	---	---	0.4	0.5
Na	0.2	1.0	0.2	0.3	1.9	0.3
Cl	0.3	0.3	0.1	0.3	1.0	0.2
S	---	0.5	0.2	0.5	0.8	0.8
P	0.5	0.4	---	---	0.3	0.1
Cu,Mg,Fe ,Zn,Ni,Cr	0.7	1.2	0.9	0.4	1.7	0.5

XPS surveys of the adhesive material that originally was in contact with the above surfaces are shown in Table V. A clean adhesive surface should show 70-75 % C, 18-24 % O, and about 5% N, with traces of Al and Si (filler). All the surfaces studied show essentially clean adhesive surfaces. Note that the high Si levels seen on the metal sides are not present on the adhesives. Normally, this indicates that the Si is in fact a silicate, since silicone readily transfers and is seen with nearly equal concentrations on both opposing sides. However, high resolution XPS, which can detect the chemical state of elements, does indicate the presence of silicone molecules. Thus surface contamination may have played a role in some failures, for example in the slick-off region of sample #2. There are very small amounts of the elements associated with dirty water that may have been transferred from the metal surfaces. The concentrations are all too low to indicate any real contamination problems that would have disrupted the bonds. Note that the relatively high level of other elements seen on sample #3 is essentially all Cr, which is a component in the coating on the core material, and may indicate a certain amount of failure within the conversion coating on the metal, so that some of the coating is transferred to the adhesive.

The overall result of the XPS investigation is that there is no clear evidence of systematic contamination that would explain a poor adhesive bond in most regions of the examined specimens. Indications of silicone, a particularly difficult contaminant, are spotty. Generally the surfaces show the expected elements, in nearly correct ratios. The metal surfaces do show evidence of tap water used for etch solution makeup and in the rinse bath. This is not recommended practice, primarily because of F that is readily adsorbed by aluminum oxide, and can be a bad actor in causing poor bond durabilities.

Table V. XPS surface concentration results from adhesive sides of surface analysis specimens (weight %)

Element	#1	#2	#3	#4	#5	#6
Al	0.7	0.9	1.8	0.4	1.0	0.9
C	70.0	70.6	61.7	69.8	70.9	69.5
O	21.8	19.8	23.9	22.2	20.9	20.0
N	6.8	6.2	5.8	4.4	5.1	6.3
F	0.1	---	1.7	---	0.6	0.7
Si	0.2	1.2	2.2	1.7	0.7	1.1
Ca	0.2	0.6	---	0.4	0.2	0.3
Na	---	0.1	---	0.3	---	0.2
Cl	---	0.1	0.2	0.1	---	0.1
S	---	0.2	0.2	0.5	0.2	0.2
P	---	0.1	0.3	---	0.3	0.1
Cu,Mg,F, Zn,Ni,Cr	---	0.2	2.2	0.2	0.1	0.6

In the absence of general contamination, and given the essentially interfacial failures seen both with SEM and XPS, there must be a reason for poor interfacial adhesion. The primary culprit in the relatively poor adhesion of adhesive to skin and rail components must be the poor (i.e., smooth) morphology of the acid etched surfaces. A better bonding pretreatment should provide surfaces that so intimately bond with the adhesive that it cannot be removed so close to the surface, but will always fail cohesively in the adhesive bulk.

4.0 Adhesive Investigation

The cure schedule used during construction of the composite panels differs significantly from that recommended by the manufacturer of the Metalbond 328 adhesive used to attach the skins to the rails and the core. The nominal 350°F, 1-2 hour long cure recommended for the adhesive was changed to 15 min. at 375°F for panel construction. The recommended heat up rate is specified at 5°F/min, but the production rate is not specified. Finally, the recommended practice is to maintain the 30-40 psi pressure used during curing until the unit temperature is below 150°F. Production practice apparently is to remove pressure at 375°F. The recommended cure schedule information, and fresh specimens of metalbond 328 adhesive used for our testing was provided by Gerald Sauer of Scitex*

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* Scitex is the new name for the company formerly known as BASF Structural Materials, 1440 North Kraemer Street, Anaheim CA, 92806, (714) 666-4363.

4.1 Glass Transition Temperature (T_g)

We used DSC to determine the glass transition temperatures of adhesive taken from various sample locations. This value is a measure of completion of cure, i.e., more completely cured adhesives will have higher T_gs. Table VI. shows the results of this study, along with samples of fresh adhesive cured according to the recommended schedule, and the modified schedule used in construction. Figures 27-32 show the actual DSC curves for the six specimens.

TableVI. Glass transition temperatures for adhesive samples taken from various test specimens and from cure cycle test pieces.

Sample	T_g (°F)
Short Beam 2A, forced debond	172
Short Beam 3, dark adhesive	179
Type I panel, "burned" adhesive	175
Type I panel, slick-off at edge	77
350°F, 1 hr	179
375°F, 15 min	165

These results raise a number of interesting points. First, the recommended cure seems to be somewhat more complete than the production cure schedule. When this adhesive is cu.ed in air at higher temperatures (>375°F) the outer surface turns brown. Thus the two specimens with dark surfaces have reached higher temperatures. They also show higher than average T_gs, although they do not exceed that of the recommended cure material. In general, the differences between the various samples are small, except for the Type I panel edge. The very low T_g seen here is more typical of the uncured adhesive. This indicates that the control of temperature distribution over the surface of the panel specimen was very poor, with one portion in the center melted by the heat, but another at the edge almost uncured.

4.2 Cure Schedule Comparison

We conducted another experiment with fresh adhesive to further examine the effect of cure on the filleting behavior of the material. Sample skin to core bonds were made by constructing small test samples using 0.063-in. thick 2024 Al skins to approximately 2 x 3 in. core specimens in a small platen press. One sample was cured according to the manufacturer's recommended schedule, i.e., 5°F/min heat up, 30 psi pressure applied, 1 hr hold at 350°F, and cool down before removal. The other was heated at 10°F/min, 30 psi applied, held for 15 min at 375°F, and pressure removed immediately, in order to simulate the production process. Both specimens were well bonded. The adhesive stayed grey , with no hint of browning at either temperature. Both specimens showed good filleting on the core. Figures 33 and 34 respectively show the core and skin sides of the test specimen bonded at 375°F after the skin was peeled off the core. Note the

significant amount of adhesive still adhering to the core, and the clean appearance of the part of the skin where the adhesive was pulled off. This test indicates that the cure procedure is not the immediate cause of poor skin to core bonding.

Contrast the surfaces of short beam sample #6 shown in Figures 35 and 36 with those of the test specimen. A band of core pull-off cells can be seen in the center of the macrographs, but even in the relatively well bonded regions at either side the amount of adhesive remaining on the core is much less than that seen above. In addition, the areas on the skin where the core pulled off the adhesive show much more remaining adhesive; the support fabric clearly seen in the test specimen skin photos is completely obscured by an adhesive layer here. This specimen had a backside skin failure and showed core buckling. Similar macrographs from a v-notch, top skin failure show exactly the same features, Figures 37 and 38. These features are not directly related to the cure schedule used, but must reflect another deficiency in bonding practice, namely inadequate pressure to force the core into the adhesive..

5.0 Discussion

A number of instances of adhesive slick-off were observed from the skin and rail surfaces. These can be attributed to two causes. In the first place the surface treatment used to prepare the skins and rails is a surface cleaner and paint pretreatment that is not intended for adhesive bonding preparation (according to MIL-C-10578D, Corrosion Removing and Metal Conditioning Compound). The lack of microporosity in the surface oxide presents a poor base for adhesion, and should prove detrimental to long-term durability. The appropriate standard for honeycomb assemblies (MIL-H-87990, Aluminum Honeycomb Sandwich Assemblies, Manufacture of) mandates phosphoric acid anodization as the only acceptable surface treatment for surface to be bonded (excluding core). Other proven surface preparation techniques such as FPL etch or P2 etch would also provide a much better bonding surface than the Oakite 33 currently used.

The second surface problem is potential contamination of the bonding surfaces. Although not found in specimens studied for this report, contamination is always a major concern in bonding operations. The current production process Fabrication Process Specification (FPS) 3.7 and 3.9 imply that the edge of the core and the rail surface on one side of the panel are sitting on the assembly table surface. This is poor bonding practice. MIL H-87990, para. 3.4.5.3.2.1 states that surfaces to be bonded should not be touched. The bonding jig design should be modified to reduce surface contact as much as possible, and careful attention paid to the cleanliness of the assembly table surface.

Poor skin to core contact (bridging) was evident in a number of locations on both test specimens and the full size panel. Platen presses typically have great difficulty maintaining an even pressure, since both the work pieces and the platens themselves are rarely perfectly flat. Some means of equalizing pressure is recommended. Silicone rubber backing sheets are typically used to spread pressure over bondments during cure. The high temperature elastomer acts to distribute pressure around high spots, and provide

even loading across the surface. A potential trouble spot with this approach must be avoided if it is pursued, however. Extremely small amounts of the silicone material will transfer from the rubber to the outer surface of the aluminum skins unless some type of separator is used. Surface contamination by silicone destroys adhesion, and therefore will cause problems with panel painting after bonding. Silicone is also extremely difficult to remove using normal cleaning techniques, like wiping with solvents. Therefore clean separator films must be used between the rubber and the panel to prevent contamination. Note also that the composite panels are relatively difficult to bond with consistent pressure, since the thick skins are much stiffer than those typically used in aircraft construction, and less able to deform around local high spots. Thus the dimensions of the rails and core must be carefully matched to avoid edge bridging. The clamping fixtures used to hold the rails during bonding must ensure that the rails are flat, since any rotation will guarantee lack of contact with the core.

The other problem related to pressure was seen in the general core to skin bonds. None of the core seemed to be sufficiently pressed into the adhesive to make normal fillets. As Figure 36 shows, the average panel core to skin bond showed considerably more adhesive underneath the honeycomb, and less in the fillet, than would be expected using standard methods. This would significantly reduce the strength of this bond, and lead to the marginal failures observed in the short beam testing. It appears that the overall pressure used to bond the mats was insufficient. Although the FPS para. 3.13 states that 35 psi should be applied to the platens, the bondments indicate that the actual pressure was lower. This certainly should be checked in the future.

Another platen press problem was the temperature control. The full size panel showed both overheated and undercured adhesive on the same skin. This strongly implies poor temperature distribution across the platens. MIL -H-87990 requires that platen temperature be measured at every point at the intersections of a 12 in. grid drawn on the platten surface, and the deviation be held to less than $\pm10°F$ if the press is to be used for bonding operations. The silicone rubber pads recommended to improve the pressure distribution might also act to reduce the temperature deviation, but it seems clear that revisions/repairs to the platen press will also be necessary to meet bonding requirements.

The short cure schedule is another reason that temperature must be well controlled. Our curing tests imply that the 15 min 375°F cure is sufficient to fully cure the adhesive, but a longer cure would be preferable to insure that complete cure occurs, especially if there are cool spots on the platens. Thermocouples should be placed on the bondment so that both hot and cool spots can be monitored. The coldest spot on the platen should be used as the control temperature monitor at the least.

Removing pressure at the end of the cure cycle before cooling is not recommended practice because the adhesive is weak and prone to creep at elevated temperatures. Pressure should be maintained on the bondment until the temperature falls below 150°F to insure that stresses in the skin do not cause local disbonds.

Another significant aspect of forming good skin to core bonds is the weight of adhesive. Since thinner adhesive forms smaller fillets, it is not recommended for skin to core bonds. The process specifications for the composite panels does not specify adhesive weight. MIL-M-52612A, Mats, Landing, Aluminum Medium Duty XM19, indicates that the adhesive weight will be 0.060-0.095 lb/ft^2. Scitex representative Gerald Sauer states that the 0.06 weight is completely inadequate for this application, and that 0.090-0.095 lb/ft^2 material should be used. Weights and areas were carefully determined from pieces of adhesive removed from the test specimens. In most cases this material had a honeycomb imprint, so that some of the adhesive material had been removed. This would mean that measured densities should be increased to account for the lost material. The measured weights for three adhesive pieces were 0.066, 0.075 and 0.081 lb/ft^2. This variation probably reflects different degrees of adhesive pull-off on the core. Assuming a 30% loss in the lowest weight case, and a 10% in the highest, the actual adhesive densities would be in the 0.090-0.095 range. Note however that the fresh "0.095" adhesive actually weighed 1.04 lb/ft^2 by our measure. It appears that a heavier weight adhesive was used to construct the test articles, but the specifications should be changed to insure that the correct adhesive is always used.

Finally, it is clear that the skin to rail bonds were almost totally absent in the test specimens and full size panel examined here. In almost every case the adhesive applied to the skin was trimmed so far away from the edges that it never reached the rails. This allowed the core splice adhesive to expand into the gap between the rail and skin, and prevented any effective bonding with the structural adhesive. The culprit here must be the "inset of the adhesive to prevent overflow on the adhesive during the curing operation" mentioned in paragraph 3.1 of the process specification. Adhesive should be applied over the entire surface of the skin, and another means of preventing overflow employed. If rubber pads and separator films are used during bonding there should be no problem of overflow, since the rubber will seal the skin to rail edges and prevent adhesive escape. High temperature adhesive tape also might be employed to seal the gaps between the skin and rail if necessary ("flashbreaker tape").

6.0 Process Improvement Plan

A number of new steps and requirements should be included in the current fabrication process specification in order to avoid repetition of the problems noted above. The main areas that clearly need improvement are the control and use of the platen press, surface preparation of the skins and rails, cleanliness of the assembly procedure, and sizing the skin adhesive. All of these points require careful attention to detail, and are therefore relatively difficult to adequately describe in a written process specification. Some, such as the addition of a bonding preparation etch or anodizing procedure to the cleaning operation, will undoubtedly add significant costs to the operation. Implementation details of such measures will best be accomplished in consultation with the manufacturer to reduce the cost impact. Another undoubtedly costly measure that might be considered would be the use of "go-by" test specimens that accompany the full scale parts through the fabrication operations, and serve as quality control checks. If properly designed so

that they truly mimic the assembly and bonding operations, such specimens can serve as early warnings to prevent the construction of substandard parts, and insure that processing errors are corrected in a timely fashion.

Suggested changes for the fabrication process specifications listed below cover the most obvious and straightforward changes needed. More comprehensive changes, such as the addition of a bonding surface treatment or the use of QC go-by specimens, are not included, since precise details are best decided jointly between the fabricator and the designer.

2.1.1 [add] MIL-H-87990-Aluminum Honeycomb Sandwich Assemblies, Manufacture of

4.0.1 [add] Platen press must conform to the requirement of MIL-H-87990.

4.0.1b Pressure mats capable of withstanding bonding temperatures and pressure shall be designed to fit between the platens and the mat sections during the bonding operations.

4.0.3 [add] The fixture shall be designed to prevent contact between the rail surfaces and work tables before adhesive has been applied.

6.2 [add] Surface preparation- (add appropriate process specifications for a suitable surface preparation for adhesive bonding, FPL, PAA, CAA)

Note: [add] All contact between skin and rail surfaces that are to be bonded and any other surface should be avoided. All assembly area surfaces should be covered with clean, replaceable materials (i.e., kraft paper) and any contact with any foreign objects avoided. Bonding fixtures must be maintained in a clean condition, and should not be allowed to be handled without gloves, and be stored in a clean area between use.

8.1 [delete] Amount of inset will be determined ... [add] Inset will not exceed 0.125 from any edge of skin.

8.6 [add] Avoid any contact with bonding surfaces while applying adhesive, setting pieces aside to cool, and assembling rails in bonding fixture.

8.12 [modify?] {Final sizing may be difficult with both skins applied when the correct amount of adhesive, i.e., no inset, is used. This procedure may have to be changed to fit to size before the first sheet is applied.}

8.13 [modify] Place the fixture and core assembly between the appropriate pressure pads, using XXXX separator films to prevent contact between the outside of the core and the surface of the pads. Place the entire assembly into the platen press for bonding. ...

8.15 [delete old, add] After the soaking period the heat should be turned off, and the assembly allowed to cool under pressure until the thermocouple attached to the fixture is below 200 degrees F. The fixture and core can then be removed and placed on a storage table for cooling.
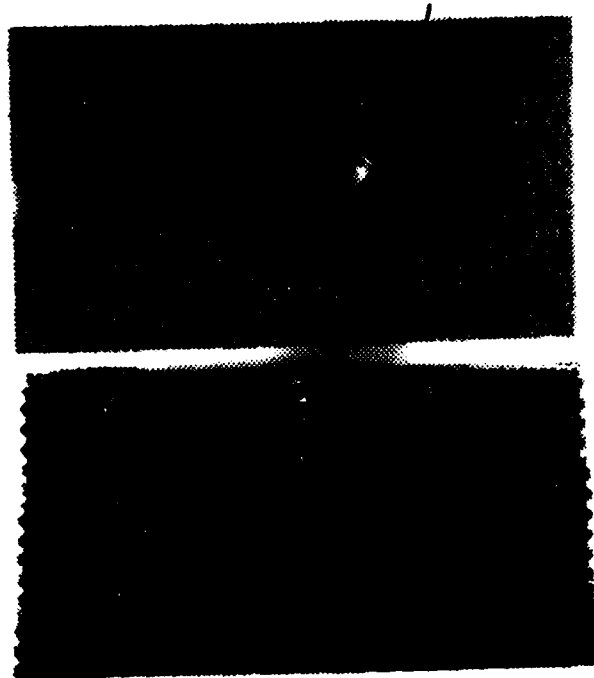
Figure 1. Short beam specimen #7, lower skin (top) and facing honeycomb (bottom). Note the lack of honeycomb imprint in the adhesive on the skin (arrow). This is a case of severe bridging.

Figure 2. Short beam specimen #1, lower skin (top) and facing honeycomb (bottom). The bumpy features on the skin adhesive (arrow) indicate extensive pull-off of adhesive from the core.
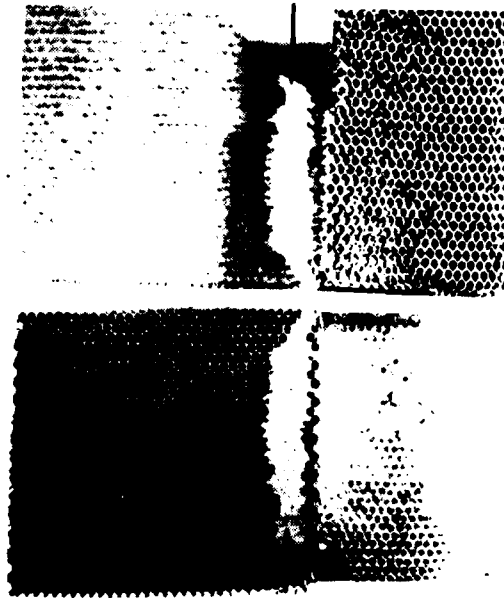
Figure 3. Upper skin (and attached core, top) and opposing core (and lower skin, bottom) of short beam specimen #2. The shiny band on the top skin, and the grey patch on the bottom core (arrows) show the adhesive-to-skin slick-off seen on this specimen.
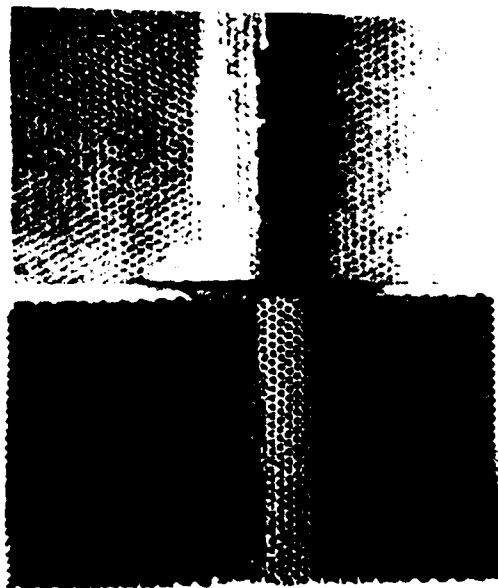
Figure 4. The upper skin (top) and opposing core (bottom) of short beam specimen #4. This specimen is typical of the "V-notch" specimens.
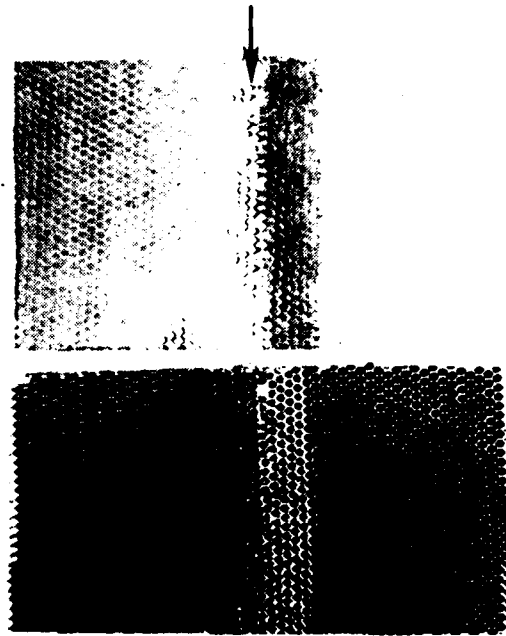
Figure 5. The upper skin (top) and opposing core (bottom) of short beam specimen #3. Another V-notch specimen; note the core pullout adhesive remnants on the skin (arrow).
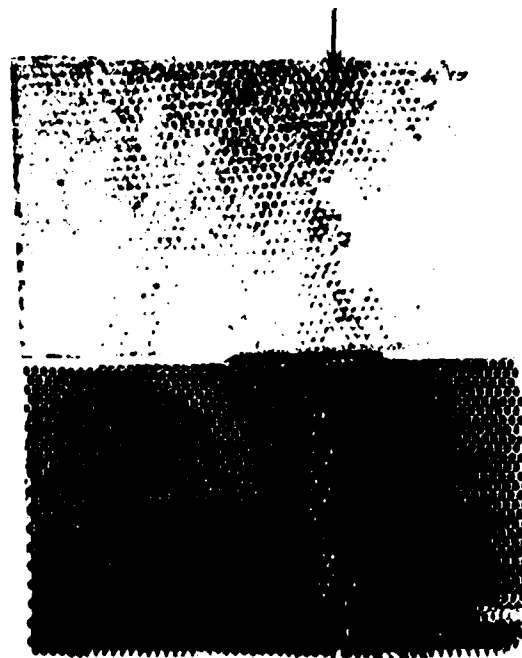
Figure 6. Bottom skin (top) and opposing core (bottom) of short beam specimen #6. Note the band of core pullout along the right side of the specimen (arrow).

Figure 7. Edge of core near a pullout region. Note the very thin adhesive fillet along some portions of the core (arrow).
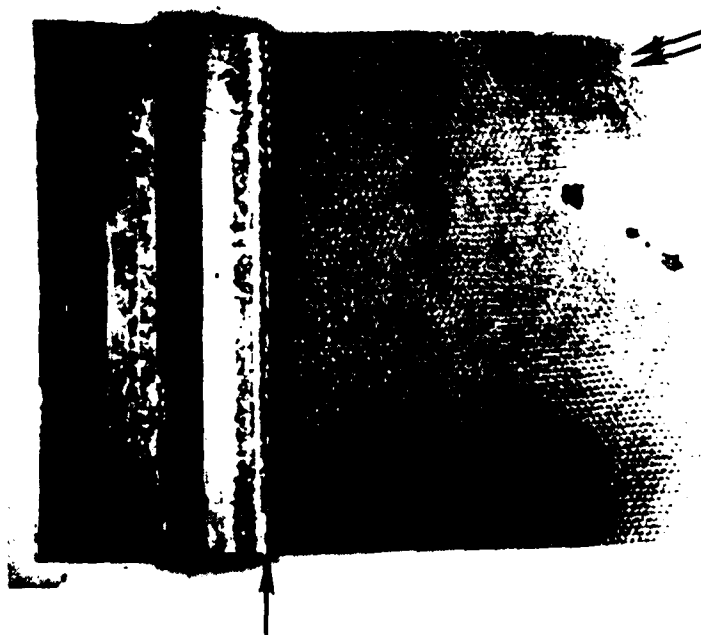
Figure 8. Upper skin and mating rail surface of static edge specimen #1. Note that skin adhesive line stops at edge of rail (arrow), and a region of bridging at upper right edge of skin (double arrow).

Figure 9. Lower skin and mating rail surface of static edge specimen #2. In this case there is adhesive covering the inner half of the rail, but there is a severe bridging problem over the core at the inner edge of the rail (arrow).
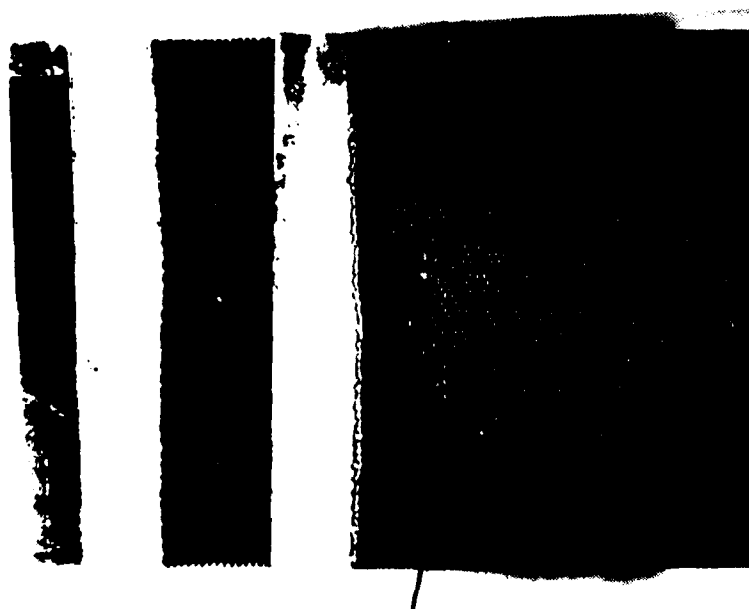
Figure 10. Skin and mating rail from static edge specimen #3. Note the curved line in the adhesive on the left side of the skin (arrow). This marks the edge of the adhesive as applied to the skin, stopping over 2 in. away from the edge.
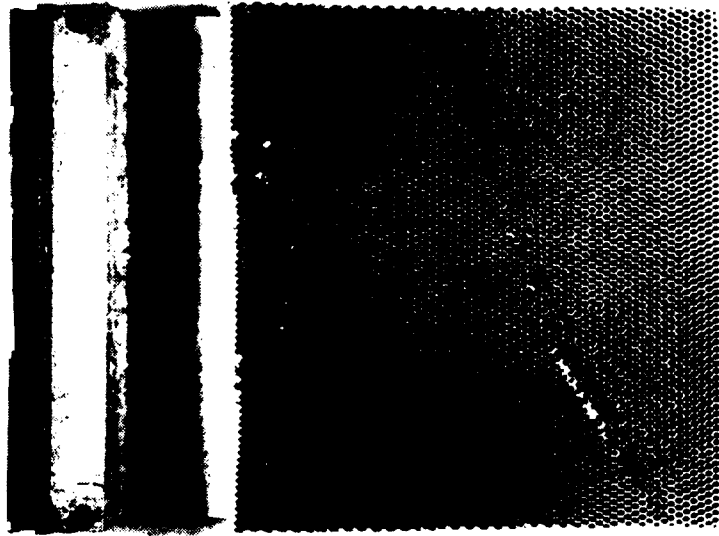
Figure 11. Rail, lower skin edge, and core from static edge specimen #4. The rail surface is coated with core splice adhesive, indicating the lack of skin adhesive over the rail.
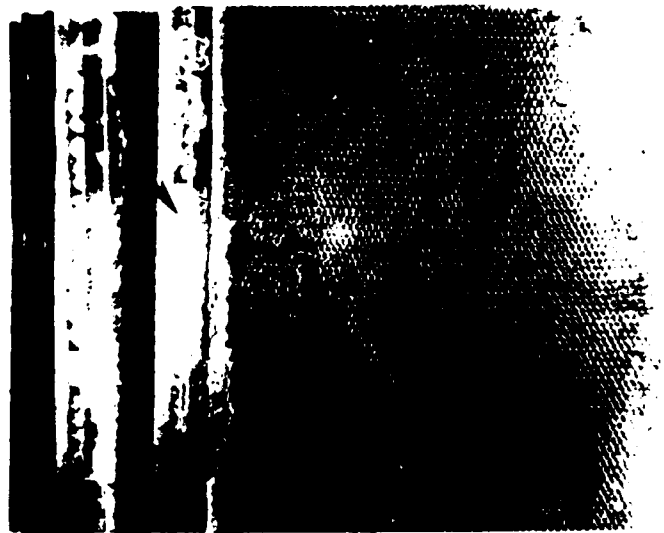
Figure 12. Skin and mating rail surface from static edge specimen #5. Extensive adhesive slick off is seen on the skin surface above the rail (arrow), and bridging in the core imprint at the lower center portion of the skin (double arrow).

Figure 13. Lower skin and mating rail section of static edge specimen #6. The line in the adhesive on the left side of the skin indicates excessive inset (arrow).

Figure 14. Lower skin from type I panel. Shiny spot in center of the panel, with tail to the right, is adhesive slick off (arrow). Spot below center is burned adhesive (double arrow).

Figure 15. Lower surface of core and rails from type I panel. Note separation and crushing of core at top of panel, and adhesive pulled off skin in the center (arrow). Also note shiny rail surfaces which had no adhesive coating (double arrow).
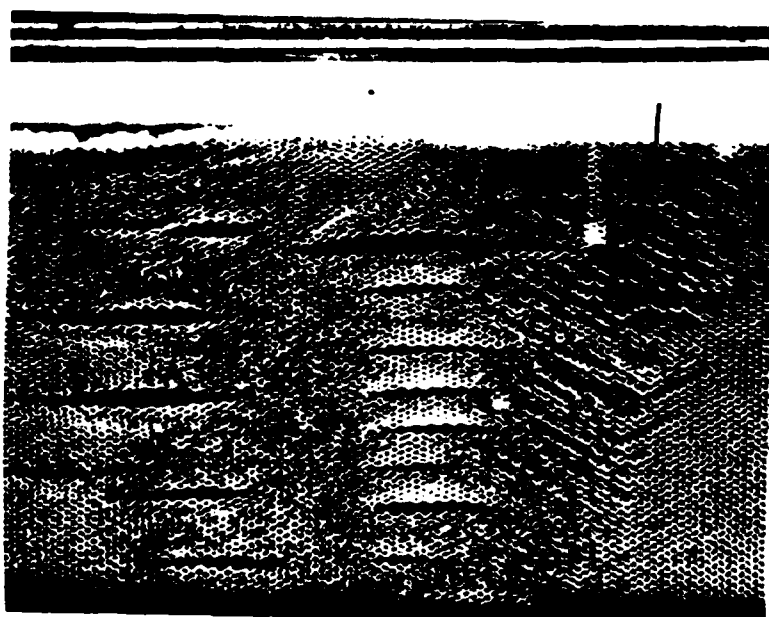
Figure 16. Close up of separated core at top of panel. Darker core region on right has very little adhesive pulled off skin, indicating poor contact (arrow).
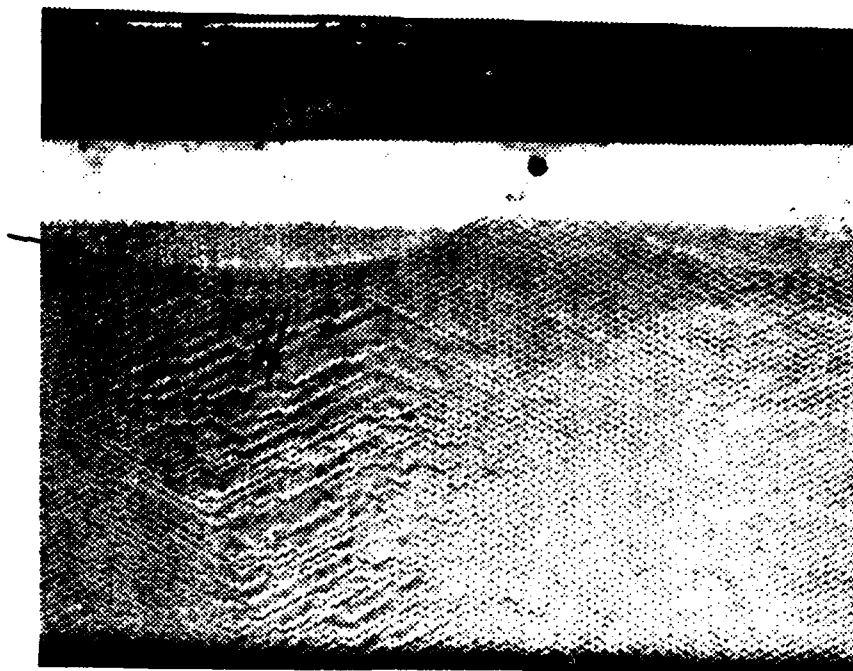
Figure 17. Close up of skin from above region. Elliptical line in adhesive edge at upper left shows excessive inset (arrow). Poor core imprint in adhesive along left side shows bridging (double arrow).

Figure 18. Close up of skin in center of panel showing slick-off above and burned adhesive below (arrow).
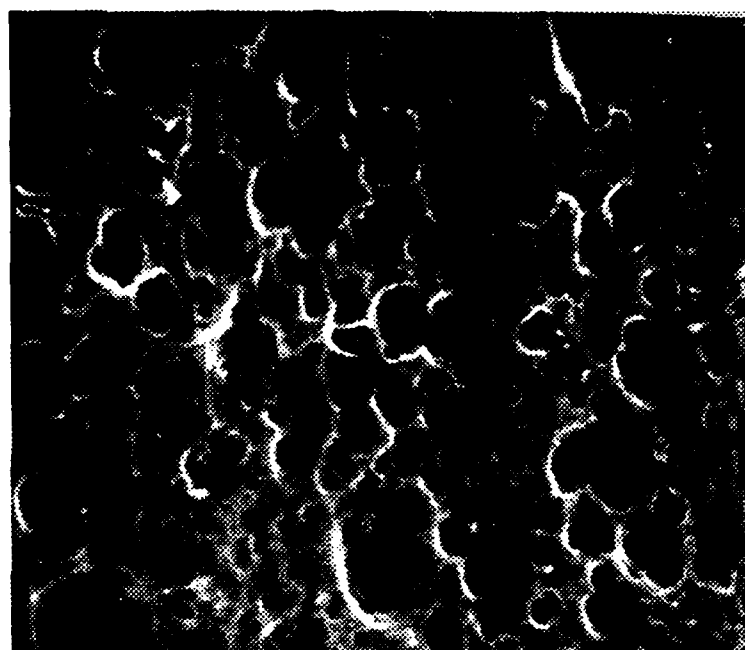
Figure 19. Electron micrograph of the metal surface of short beam specimen #2 in the slick-off region. The "lunar landscape" is typical of an acid-etched aluminum surface.
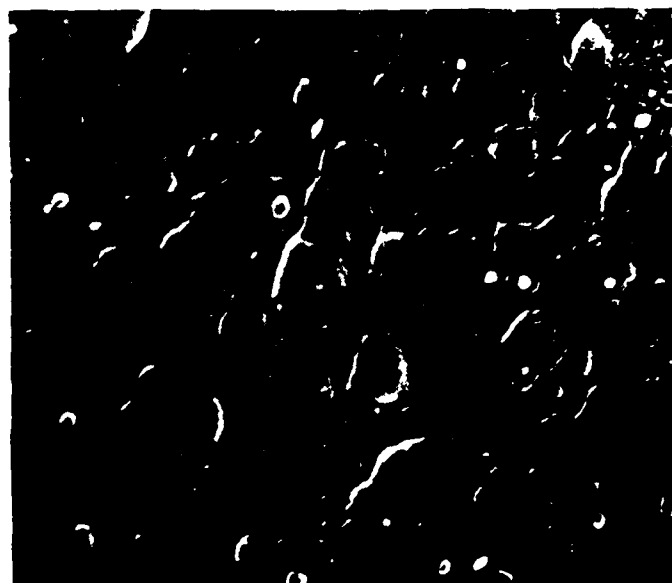
Figure 20. Electron micrograph of the adhesive surface opposing the metal shown in Figure 19. The surface exactly replicates the details of the metal side.
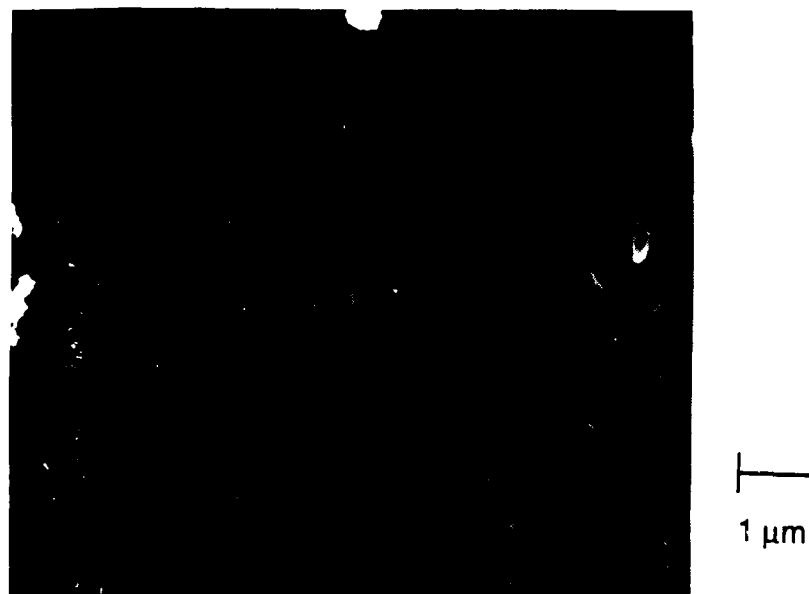
Figure 21. Electron micrograph of the edge of the core in a disbond region of short beam specimen # 4. The smooth surface with shallow lines is typical of conversion coating on aluminum core.
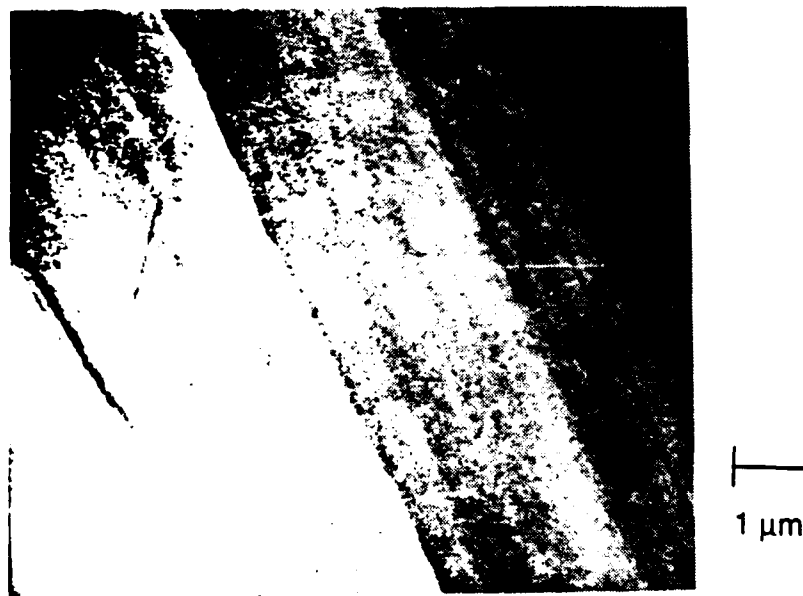
Figure 22. Electron micrograph of the adhesive pulled from core in Figure 21. Again the adhesive has replicated the metal/oxide surface, indicating a clean break at the interface.
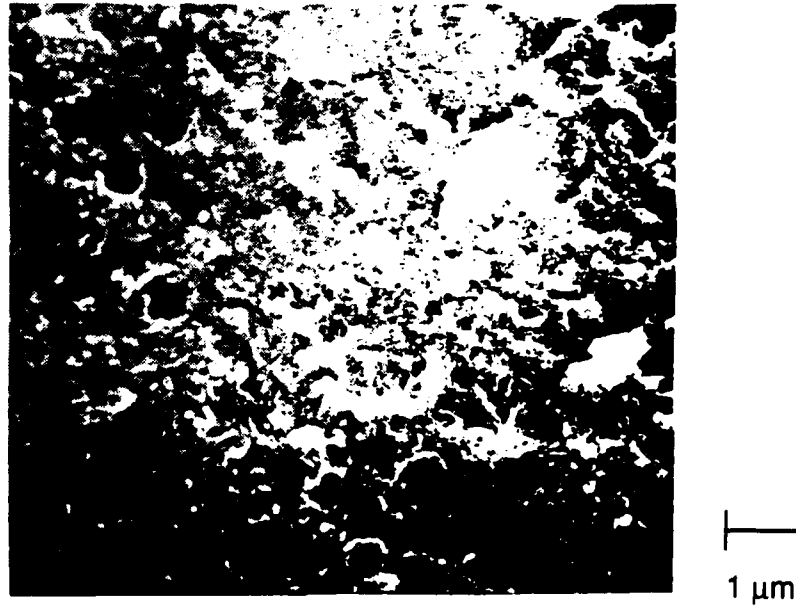
Figure 23. Electron micrograph of the skin surface of static edge specimen #2 in a slick-off region. In this case the etched aluminum morphology is covered by a layer of adhesive.
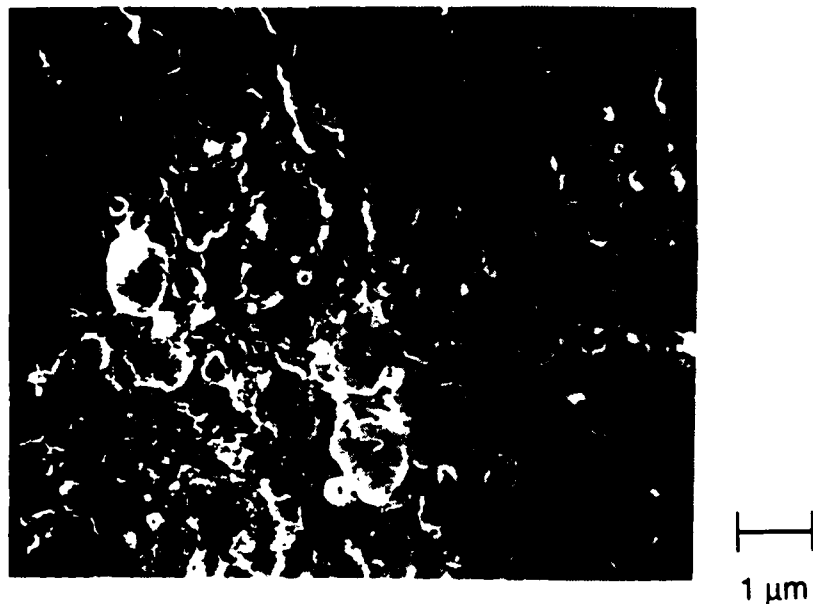
Figure 24. Mating surface of figure 22. Here the surface reflects the rougher, cohesive failure surface of the metal side.

1 μm
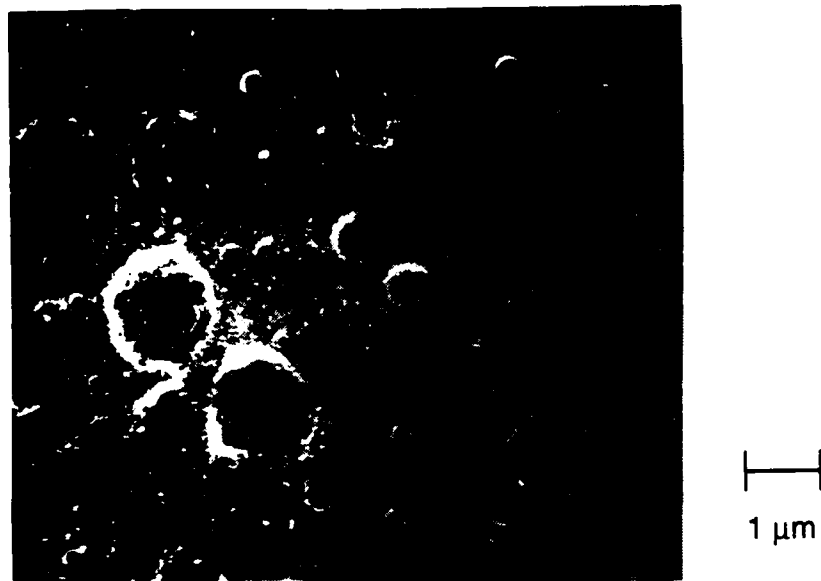
Figure 25. Electron micrograph of the skin surface on the type I panel in a slick-off region at the edge (part of the tail from the central slick-off). The etched surfaced appears to be coated with a light layer of adhesive.

1 μm
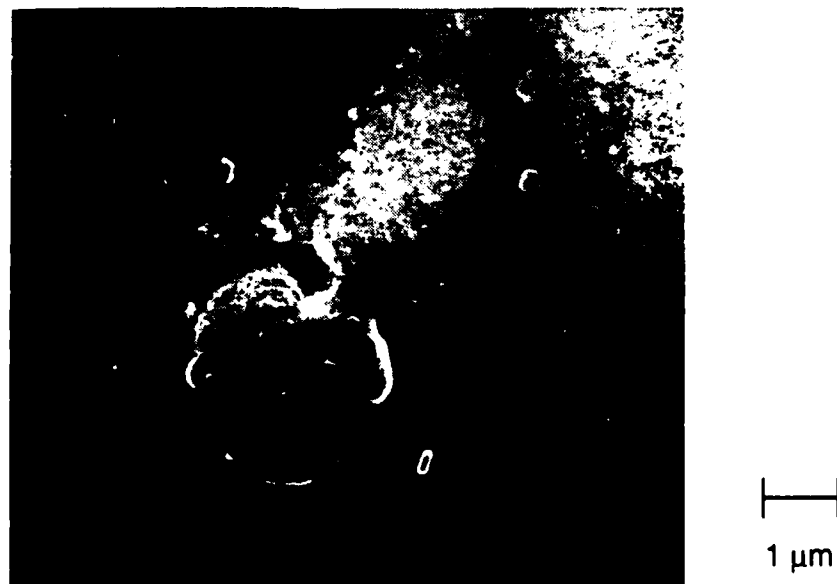
Figure 26. Electron micrograph of the adhesive mating surface of the edge of the type I panel. This surface lacks the sharp-edged features of true replicas, indicating a cohesive rather than interfacial failure. DSC of adhesive material taken from this region indicate lack of cure, which would explain a cohesive failure.
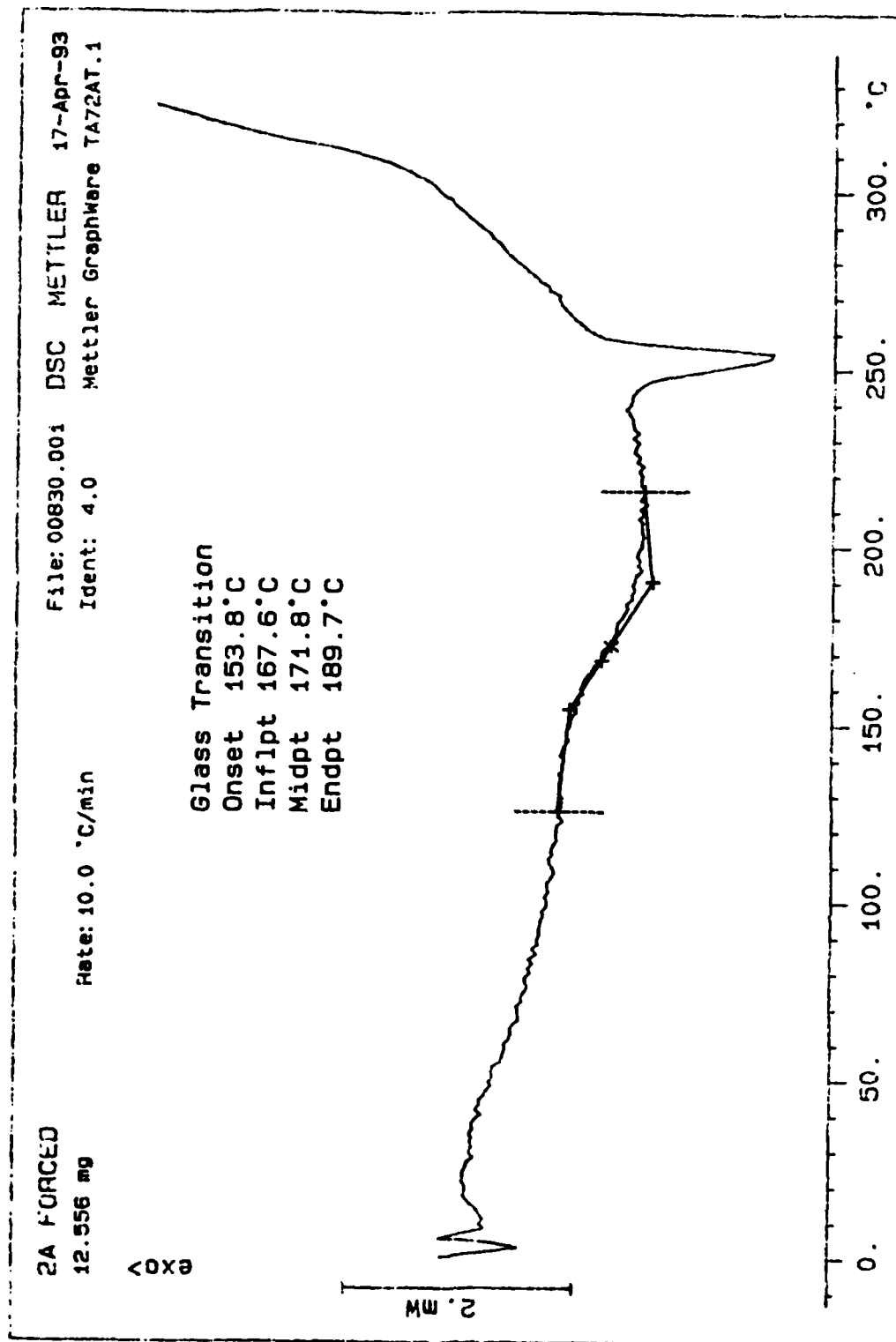
Figure 27. DSC trace of a piece of adhesive removed from short beam sample #3.
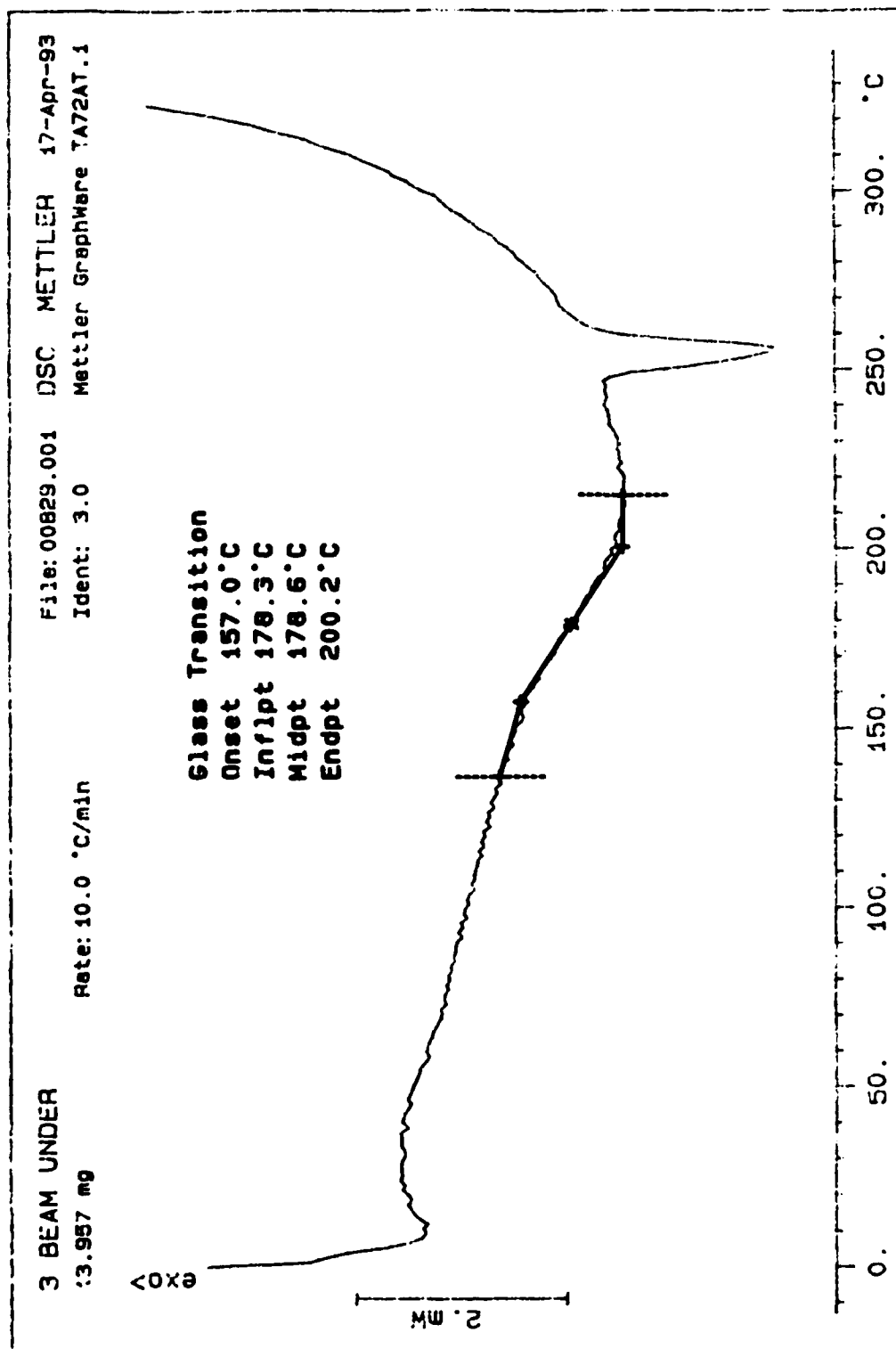
Figure 28. DSC trace of a piece of adhesive removed from short beam sample #6

LARGE CENTER BROWN

11.395 mg   Rate: 10.0 °C/min

File: 00828.001   DSC METTLER   17-Apr-93

Ident: 2.0   Mettler GraphWare TA72AT.1

exo>

2. mW

Glass Transition
Onset   156.0°C
Inflpt 160.9°C
Midpt   175.4°C
Endpt   194.8°C

0.   50.   100.   150.   200.   250.   300.   °C
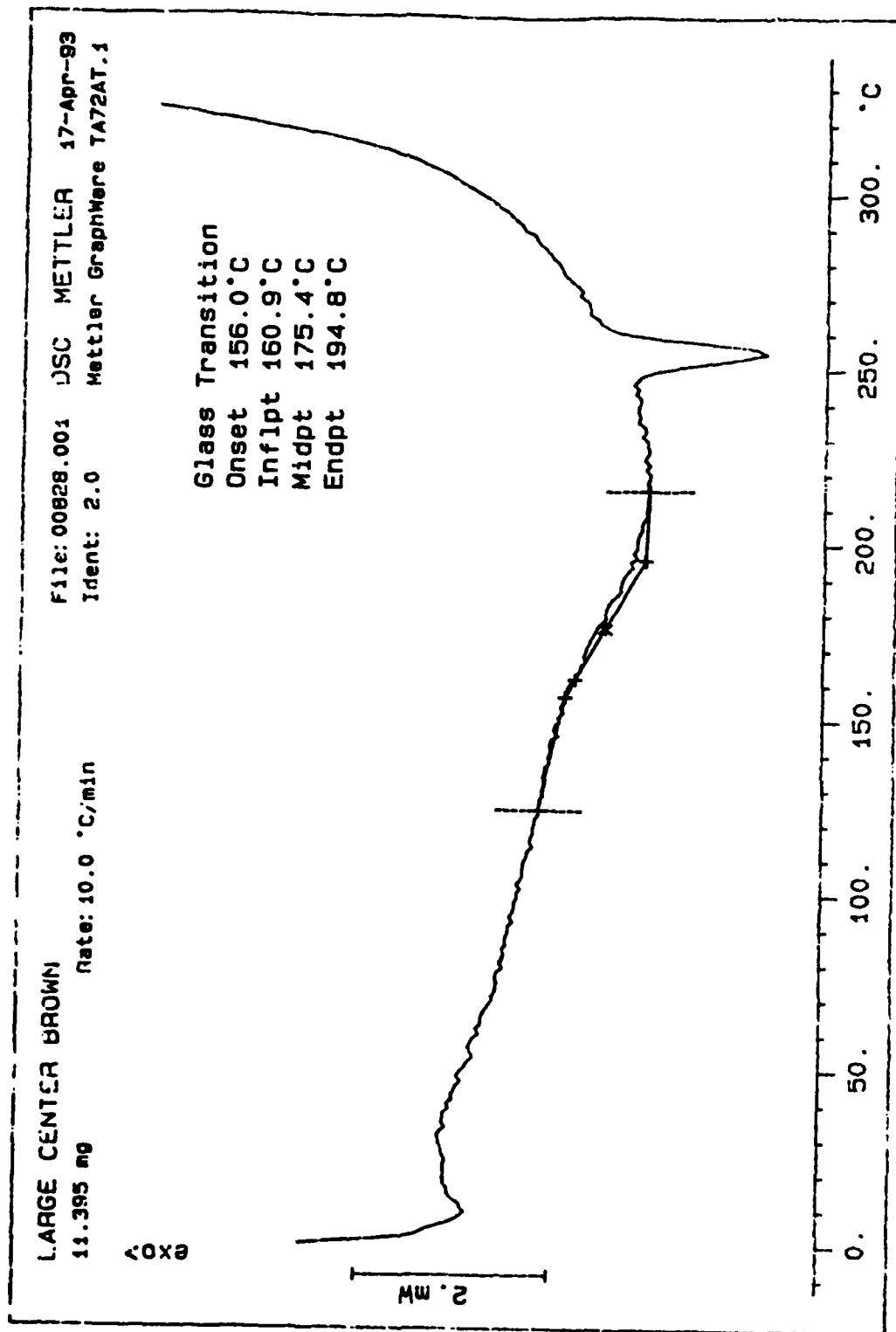
Figure 29. DSC trace of a piece of adhesive removed from the center of the type I panel (brown).
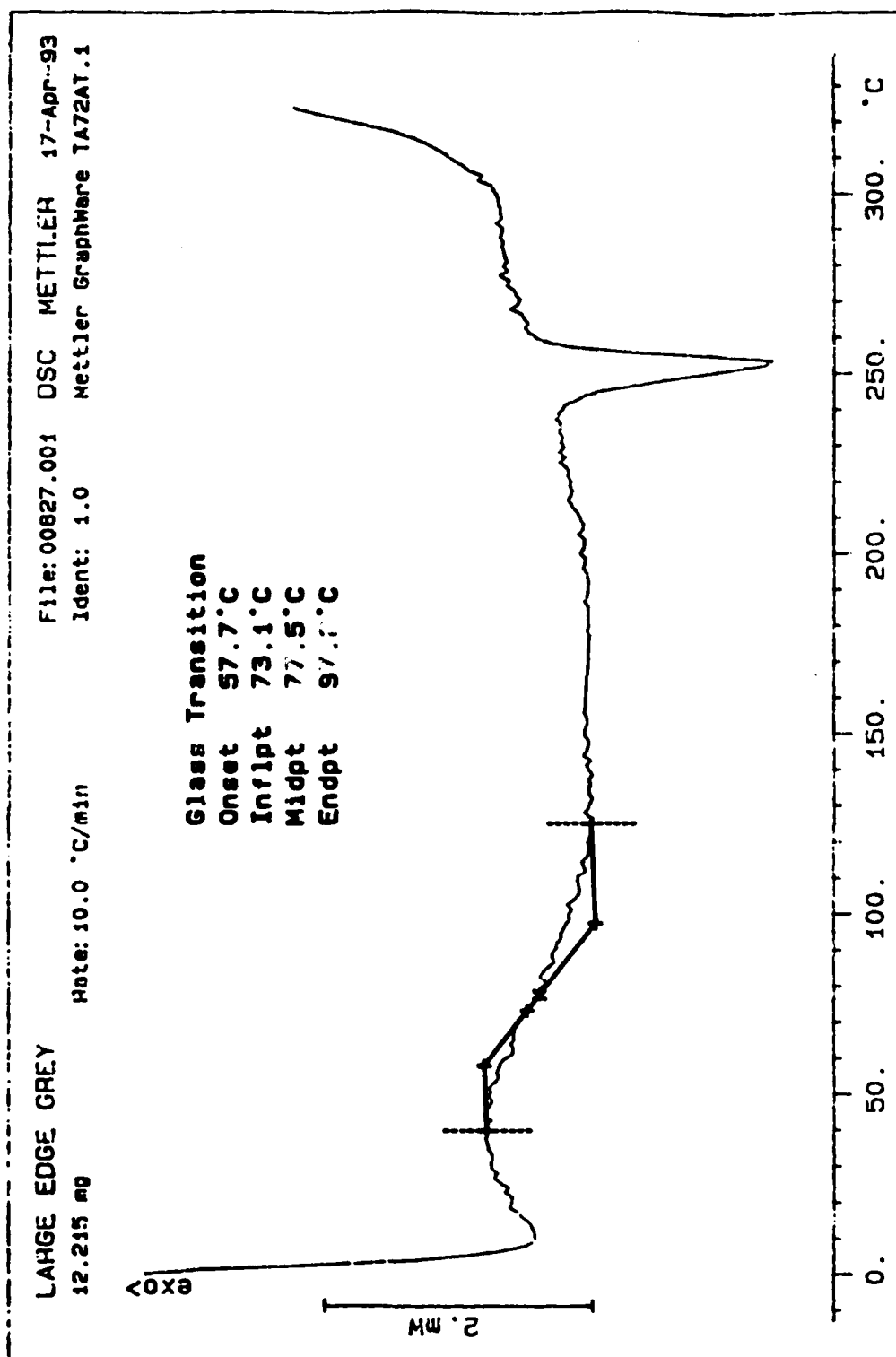
Figure 30. DSC trace of a piece of adhesive removed from the edge of the type I panel (slick-off).
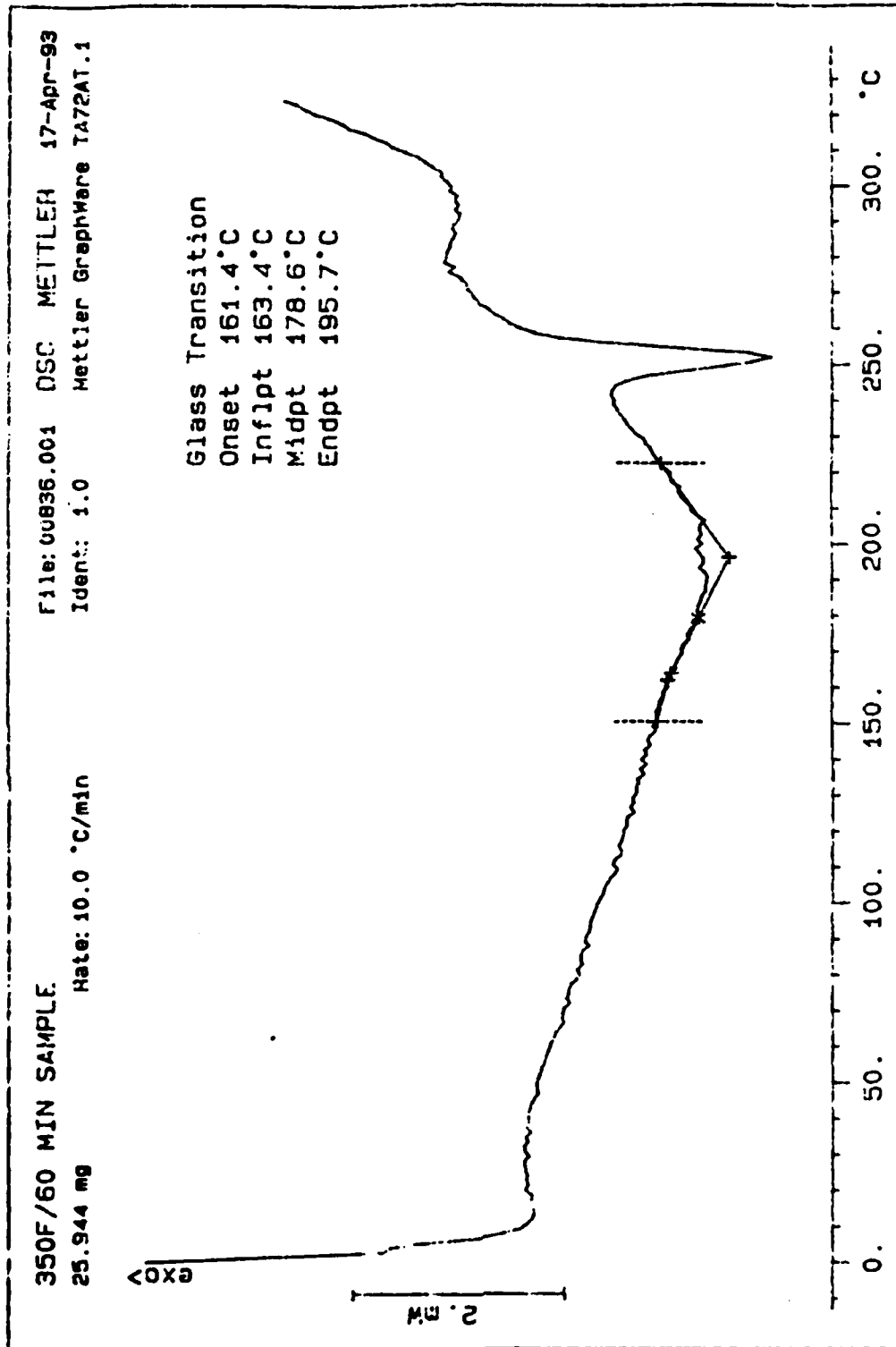
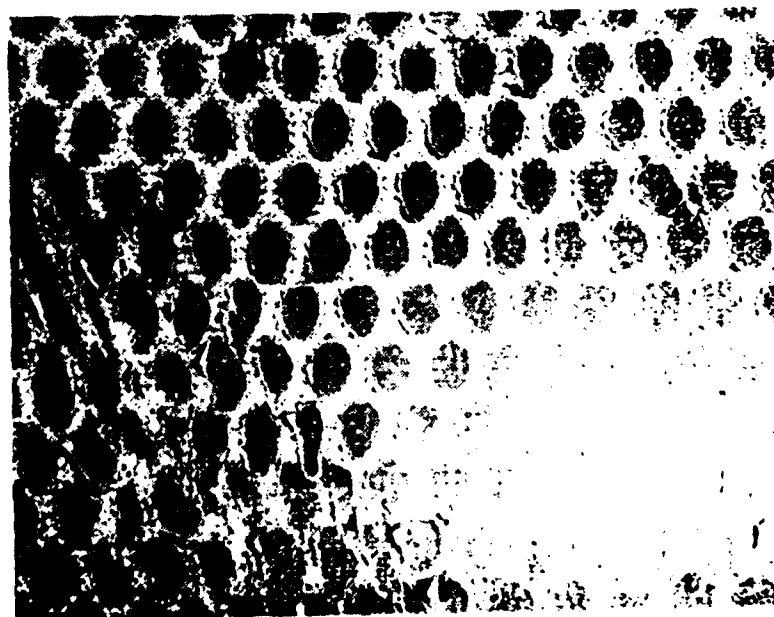Figure 31. DSC trace of adhesive that was cured for one hour at 350°F.
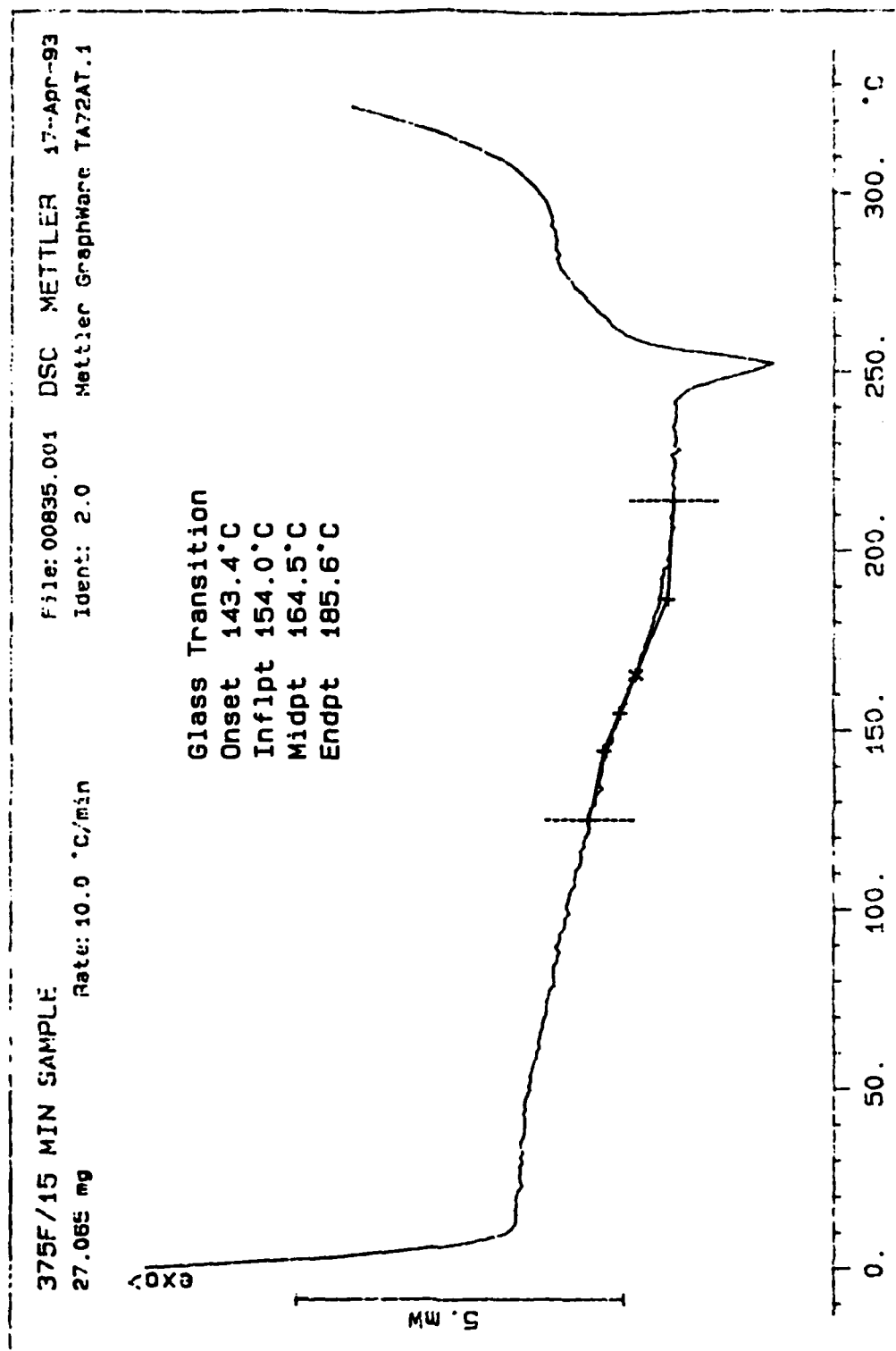
Figure 32. DSC trace of adhesive that was cure for 15 minutes at 375°F.
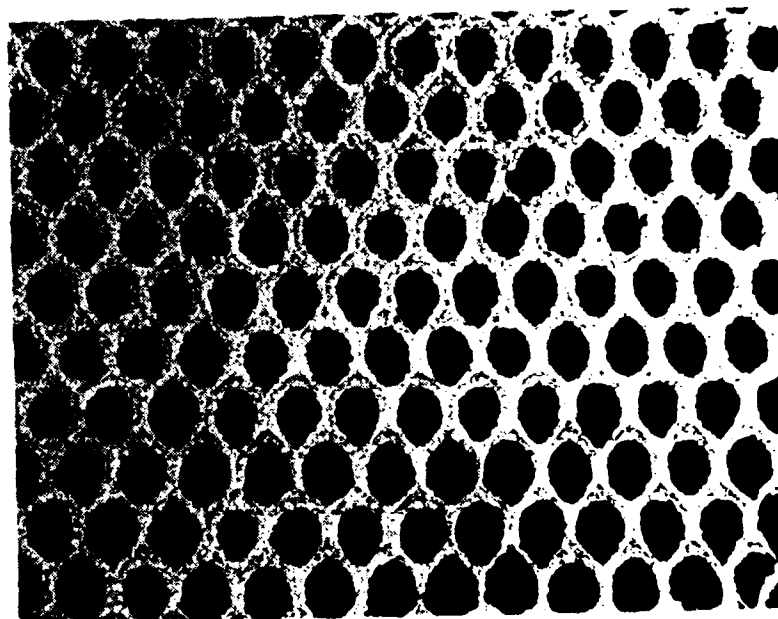
Figure 33. Macrograph of the core of the 375°F-cured test sample. Note the extensive adhesive fillet on all edges of the core.

Figure 34. Macrograph of the skin side of the 375°F-cured test sample. Support fibers can clearly be seen in regions where the honeycomb pulled off adhesive.
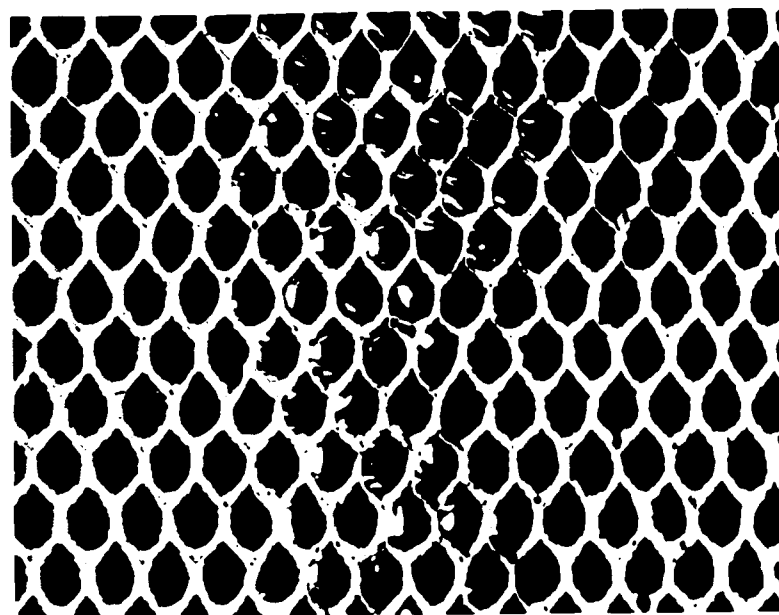
Figure 35. Macrograph of the core side of short beam sample #6. Relatively little adhesive remains on the core.
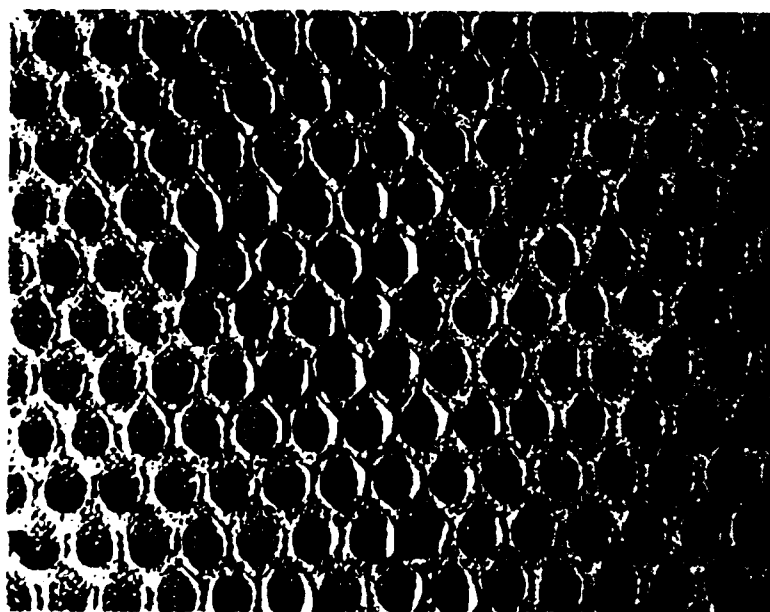

Figure 36. Macrograph of the skin side of the short beam sample #6. Adhesive still coats the support fibers under the core imprint.
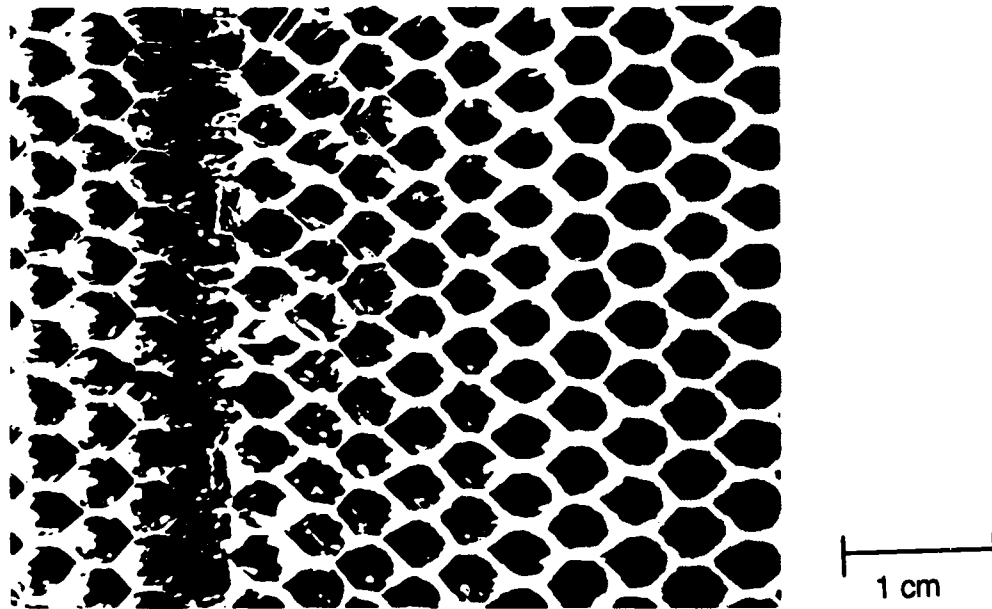
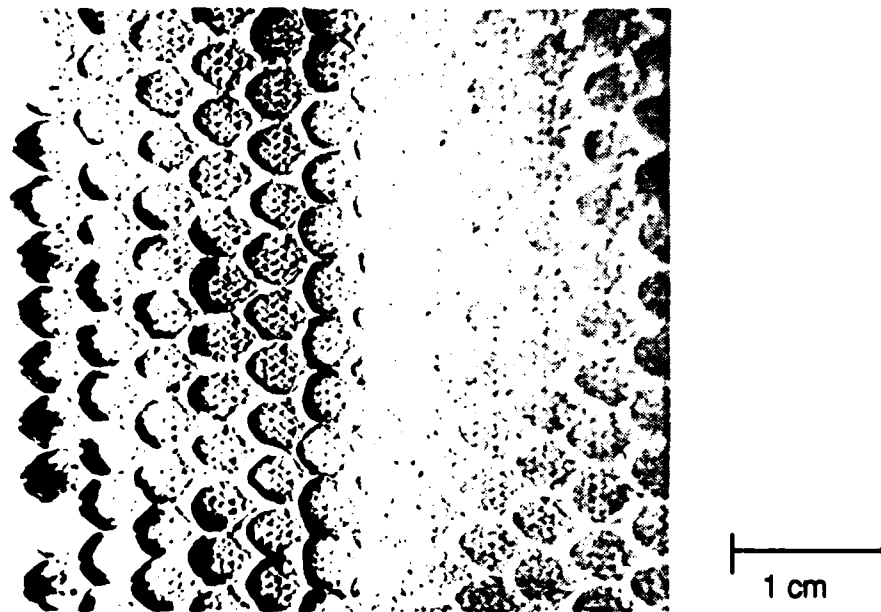Figure 37. Macrograph of the core side of short beam sample #3.

Figure 38. Macrograph of the core side of short beam sample #3.